FORM PTO-1390 (REV. 10-2000) U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE	ATTORNEY'S DOCKET NUMBER
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371	P0557/7045 DRW
	U S APPLICATION NO (If known, see 37 CFR 1.5) 09/889610

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/AU00/00018	17 January 2000 (17.01.00)	18 January 1999 (18.01.99)

TITLE OF INVENTION
USE OF VEGETATIVE MATERIAL AS A FILLER IN COMPOSITE MATERIALS

APPLICANT(S) FOR DO/EO/US
HAJI AHMAD, Khalid , SIVASITHAMBARAM PILLAI MAILVAGANAM PILLAI, Mailvaganam Thavalingam, VETTIVALOO ARUNASALAM, Arulgnanam

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to promptly begin national examination procedures (35 U.S.C. 371(f)).
4. ☐ The US has been elected by the expiration of 19 months from the earliest claimed priority date (PCT Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2)).
   a. ☒ is transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☐ has been transmitted by the International Bureau.
   c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
   a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☐ has been transmitted by the International Bureau.
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)).
   a. ☐ are attached hereto (required only if not transmitted by the International Bureau).
   b. ☐ have been communicated by the International Bureau.
   c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
   d. ☒ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(C)(5)).

Items 11. To 16. Below concern document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A FIRST preliminary amendment.
14. ☐ A SECOND or SUBSEQUENT preliminary amendment.
15. ☐ A substitute specification.
16. ☐ A change of power of attorney and/or address letter.
17. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821-1.825.
18. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
19. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
20. ☒ Other items or information:
    Copy of PCT Published Application
    Copy of International Preliminary Examination Report
    Copy of PCT/IPEA/402

Express Mail Label No. EL819464696US
Date Mailed: 18 July 2001

U.S. APPLICATION NO (If known, see 37 C F R 1 5)	INTERNATIONAL APPLICATION	ATTORNEY'S DOCKET NUMBER
09/889610	PCT/AU00/00018	P0557/7045 DRW

21. ☐ The following fees are submitted:

BASIC NATIONAL FEE (37 CFR 1.492(a)(1)-(5)):		CALCULATIONS PTO USE ONLY
Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO	$1000.00	
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO	$860.00	
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but but international search fee paid to USPTO (37 CFR 1.445(a)(2)).paid to USPTO	$710.00	
International preliminary examination fee paid to USPTO (37 CFR 1.482) But all claims did not satisfy provisions of PCT Article 33(1)-(4)	$690.00	
International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1)-(4)	$100.00	
ENTER APPROPRIATE BASIC FEE AMOUNT =		

Surcharge of $130.00 for furnishing the oath or declaration later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492(e)).				$1000.00	
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total Claims	37 - 20 =	17	X $18.00	$306.00	
Independent Claims	8 - 3 =	5	X $80.00	$400.00	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+$270.00	$	
TOTAL OF ABOVE CALCULATIONS			=	$1,706.00	
☒ Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by ½.				$853.00	
SUBTOTAL			=	$853.00	
Processing fee of $130.00 for furnishing the English translation later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				$	
TOTAL NATIONAL FEE			=	$853.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate coversheet (37 CFR 3.28, 3.31). $40.00 per property			+	$	
TOTAL FEES ENCLOSED			=	$853.00	
				Amount to be: refunded	$
				charged	$

a. ☒ A check in the amount of $1,353.00 to cover the above fees is enclosed.

b. ☐ Please charge my Deposit Account No. _____ In the amount of $_____ To cover the above fees. A duplicate copy of this sheet is enclosed.

c. ☒ The commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 23/2825. A duplicate of this sheet is enclosed.

d. ☐ Fees are to be charged to a credit card. WARNING: Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO

WOLF, GREENFIELD & SACKS, P.C.
600 Atlantic Avenue
Boston, Massachusetts 02210
Tel: (617) 720-3500

CUSTOMER NUMBER 23628

SIGNATURE

Douglas R. Wolf
NAME

36,971
REGISTRATION NO

Attorney's Docket No. P0557/7045 DRW
Express Mail Label No.: EL819464696US
Date of mailing: July 18, 2001

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant:	Contract Research & Development (M) Sdn. Bhd. and David John Tadgell
Serial No.:	Not yet assigned
Filing date:	Herewith
For:	USE OF VEGETATIVE MATERIAL AS A FILLER IN COMPOSITE MATERIALS
Examiner:	Not yet assigned
Art Unit:	Not yet assigned

Box PCT
Commissioner for Patents
Washington, D.C. 20231

Sir:

PRELIMINARY AMENDMENT

Please amend the above-identified application, prior to Examination on the merits, to conform the specification with U.S. practice:

In the Specification

On page 1, before the first paragraph, please add the subheading:

-- Field of the Invention--.

On page 1, after the subheading "Field of the Invention," please insert the following paragraph:

--This application is a continuation of application no. PCT/AU00/00018, filed on January 17, 2000 which claims priority to AU PP 8198 filed January 18, 1999, both of which are hereby incorporated by reference in their entirety.--.

On page 1, line 17, please add the subheading:

--Background of the Invention--.

In the Abstract

Please add the following Abstract (also attached on a separate sheet at the end of this preliminary amendment):

--This invention relates to the use of a filler derived from cereal husk, more particularly rice husk, in composite materials to enhance the flame retardant, antistatic, accelerator, plasticiser and blowing characteristics in various composite materials.--.

In the Claims

Please rewrite claims 5, 6, 7, 9, 10, 11, 14, and 16 as follows:

5. (Amended) A process according to claim 3 wherein said process utilizes fresh rice husk as the vegetative material.

6. (Amended) A filler according to claim 1 when produced by a process comprising burning a fresh vegetative-based material, ground to a particle size from 100 mesh to 400 mesh, at about 803° to 804° C for 3 to 4 seconds.

7. (Amended) A method for improving the anti-static, flame retardant, accelerator, plasticiser and/or blowing characteristics of a composite material wherein said method comprises blending into the composite material with a carbonized vegetative-based filler according to claim 1 and wherein said blending is substantially completed prior to incorporation of any additives, if any.

9. (Amended) A method according to claim 7 wherein the composite material is latex (NR/SR) the dosage of the carbonized vegetative filler is from 1.5 to 2.5 phr (parts per hundred).

10. (Amended) A method according to claim 7 wherein said composite material is selected from the group comprising:

i) thermoplastic resins;

ii) thermoset plastics;

iii) rubbers and elastomeric materials;

iv) conductive coatings;

v) printing inks;

vi) bitumen; and

vii) concrete.

11. (Amended) A composite material having improved anti-static, flame retardant, accelerator, plasticiser and/or blowing characteristics wherein said composite material is produced by the method of claim 7.

14. (Amended) A method according to claim 12 wherein the rice husk and tyre crumb is mixed in composition with a palm oil effluent prior to it being added to the bitumen.

16. (Amended) A method according to claim 14 wherein the composition is added in a dosage amount of about 20% by weight of the said bitumen.

On page 22, please delete lines 23-29.

REMARKS

In view of the foregoing amendments, consideration and favorable action are respectfully requested.

Respectfully submitted,

By: _____
Douglas R. Wolf, Reg. No. 36,971
WOLF, GREENFIELD & SACKS, P.C.
600 Atlantic Avenue
Boston, MA 02210
(617) 720-3500

Attorney's Docket No.: P0557/7045 DRW
Date: July 18, 2001

MARKED-UP CLAIMS

5. (Amended) A process according to claim 3 [or claim 4] wherein said process utilizes fresh rice husk as the vegetative material.

6. (Amended) A filler according to [any one of claims] claim 1 [or 2] when produced by a process [according to claim 4] comprising burning a fresh vegetative-based material, ground to a particle size from 100 mesh to 400 mesh, at about 803° to 804° C for 3 to 4 seconds.

7. (Amended) A method for improving the anti-static, flame retardant, accelerator, plasticiser and/or blowing characteristics of a composite material wherein said method comprises blending into the composite material with a carbonized vegetative-based filler according to claim 1 [or clam 2] and wherein said blending is substantially completed prior to incorporation of any additives, if any.

9. (Amended) A method according to claim 7 [or claim 8] wherein the composite material is latex (NR/SR) the dosage of the carbonized vegetative filler is form 1.5 to 2.5 phr (parts per hundred).

10. (Amended) A method according to [any one of claims] claim 7 [to 9] wherein said composite material is selected from the group comprising:

i) thermoplastic resins;
ii) thermoset plastics;
iii) rubbers and elastomeric materials;
iv) conductive coatings;
v) printing inks;
vi) bitumen; and
vii) concrete.

MARKED-UP CLAIMS

11. (Amended) A composite material having improved anti-static, flame retardant, accelerator, plasticiser and/or blowing characteristics wherein said composite material is produced by the method of [any one of claims] claim 7 [to 10].

14. (Amended) A method according to claim 12 [or claim 13] wherein the rice husk and tyre crumb is mixed in composition with a palm oil effluent prior to it being added to the bitumen.

16. (Amended) A method according to claim 14 [or claim 15] wherein the composition is added in a dosage amount of about 20% by weight of the said bitumen.

ABSTRACT

This invention relates to the use of a filler derived from cereal husk, more particularly rice husk, in composite materials to enhance the flame retardant, antistatic, accelerator, plasticiser and blowing characteristics in various composite materials.

Attorney's Docket No. P0557/7045 DRW
Express Mail Label No.: EL819464696US
Date of mailing: July 18, 2001

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant:	Contract Research & Development (M) Sdn. Bhd. and David John Tadgell
Serial No.:	Not yet assigned
Filing date:	Herewith
For:	USE OF VEGETATIVE MATERIAL AS A FILLER IN COMPOSITE MATERIALS
Examiner:	Not yet assigned
Art Unit:	Not yet assigned

Box PCT
Commissioner for Patents
Washington, D.C. 20231

Sir:

STATEMENT FILED PURSUANT TO THE DUTY OF DISCLOSURE UNDER 37 CFR §§1.56, 1.97 AND 1.98

Pursuant to the duty of disclosure under 37 C.F.R. §§1.56, 1.97 and 1.98, the Applicants request consideration of this Information Disclosure Statement.

PART I: Compliance with 37 C.F.R. §1.97

This Information Disclosure Statement is being filed with a National Application. No fee or certification is required.

PART II: - Information Cited

The Applicants hereby make of record in the above-identified application the information listed on the attached form PTO-1449 (modified). The order of presentation of the references should not be construed as an indication of the importance of the references.

The Applicants hereby make the following additional information of record in the above-identified application:

The Examiner's attention is hereby drawn to the International Search Report mailed February 6, 2001 in PCT Application PCT/AU00/00018, filed January 17, 2000, which corresponds the patent application filed herewith.

PART III: Remarks

A copy of each of the above-identified information is enclosed unless otherwise indicated on the attached form PTO-1449 (modified). It is respectfully requested that:

1. The Examiner consider completely the cited information, along with any other information, in reaching a determination concerning the patentability of the present claims;

2. The enclosed form PTO-1449 be signed by the Examiner to evidence that the cited information has been fully considered by the Patent and Trademark Office during the examination of this application;

3. The citations for the information be printed on any patent which issues from this application.

By submitting this Information Disclosure Statement, the Applicants make no representation that a search has been performed, of the extent of any search performed, or that more relevant information does not exist.

By submitting this Information Disclosure Statement, the Applicants make no representation that the information cited in the Statement is, or is considered to be, material to patentability as defined in 37 C.F.R. §1.56(b).

By submitting this Information Disclosure Statement, the Applicants make no representation that the information cited in the Statement is, or is considered to be, in fact, prior art as defined by 35 U.S.C. §102.

Notwithstanding any statements by the Applicants, the Examiner is urged to form his own conclusion regarding the relevance of the cited information. An early and favorable action is hereby requested.

Respectfully submitted,

By: _____

Douglas R. Wolf, Reg. No. 36,971
WOLF, GREENFIELD & SACKS, P.C.
600 Atlantic Avenue
Boston, MA 02210
(617) 720-3500

Attorney's Docket No.: P0557/7045 DRW
Date: July 18, 2001

09/889610 PCT/AU00/00078
Received 09 October 2000

 Rec'd PCT/PTO 18 JUL 2001

USE OF VEGETATIVE MATERIAL AS A FILLER IN COMPOSITE MATERIALS

TECHNICAL FIELD

This invention relates to the use of a filler derived from cereal husk, more particularly rice husk, in composite materials to enhance the flame retardant, antistatic, accelerator, plasticiser and blowing characteristics in various composite materials. The invention has particular but not exclusive application to the following families of composites:-

1. Thermoplastic Resins
2. Thermoset Plastics
3. Rubbers and Elastomeric Materials
4. Conductive Coatings and Printing Inks
5. Bitumen
6. Concrete

BACKGROUND ART

Composite materials are well known. Fillers are usually added to composite materials, including composite polymers, to save cost or to enhance a particular mechanical property or other characteristic of the materials. The usage of fillers is usually accompanied by coupling agents that enhance the polymer-filler and filler-filler interaction so that the expected properties are realised.

The present invention is concerned with fillers which enhance the anti-static, flame retardant, accelerator, plasticiser, blowing characteristic and/or other physical or mechanical properties of composite materials and has particular application for use in composite polymers. Such have wide application in the aeronautical, mining, computer, road building, textile, foot ware, rubber and polyurethane industries among others. For example, it is often desirable to prevent the build up of static charges which can cause sparks (and hence explosions or electrical damage) or production problems, eg. collection of dust and poor feeding of materials through machinery. More highly conductive composite polymers can also be used for Electro Magnetic Interference shielding, for example.

Carbon black fillers, aluminium flakes and fibres, stainless steel fibres and chopped carbon fibres have all been used as fillers for the purpose of rendering composite plastic conductive. Likewise other chemicals such as Halogen compounds or triethyl phosphate have been used to achieve the flame retardant property.

A number of theories have been proposed to explain how discreet particle fillers impart conductivity and flame retardant properties in composite plastics.

In order for current to flow in a conductive polymer compound, electrons must travel along the filler as the plastic itself is an excellent insulator. To achieve this flow the discreet particles of the filler must be in contact or separate by a minimum distance which is probably less than 100 Angstroms. There are three properties of the filler particles which will effect the average inter-particle distance for a given filler loading in a polymer system. These are particle size, shape (structure), and porosity. Smaller size, irregular shape and high porosity all result in smaller inter-particle distances and hence higher conductivity. A fourth property of the particle which is relevant to conductivity and flame retardant properties in the composite plastics is surface chemistry, that is the presence of oxygen on the surface. The presence of appreciable quantities of oxygen on the surface (called volatile content) acts as insulation and hence reduces conductivity.

The known conductive fillers such as carbon black, aluminium, stainless steel and carbon fibres are expensive and furthermore some of these materials have other processing difficulties, eg. aluminium fibres and stainless steel fibres settle in liquid environments due to their high density. Further problems with known conductive fillers are that they often compromise other properties of composite plastics such as flame retardance and strength.

Static electrification of articles can lead to a number of undesirable effects including:

- Attraction of dust particles.
- Attraction between surfaces, e.g. plastic films and textile yarns.
- Risk of fire or explosion caused by sparking near inflammable liquids, gases, and explosive dusts, e.g. coal dust and flour.
- Risk of shock to persons handling equipment.

The accumulation of electrostatic charges can be prevented by using materials of low resistance. The resistivity of natural rubber can be lowered by compounding with suitable ingredients. Alternatively, as static electrification is a surface phenomenon, the product can be covered with a conducting surface layer.

Low resistance rubber is required for a wide range of applications, such as rollers for textile machinery, conveyor belting, fuel hoses, flooring, footwear, antistatic gloves (electronic industry), cables, equipment used in hospital operating theatres, and aircraft components.

The terms "antistatic" and "conductive" are restricted here to rubber products rather than the rubber itself because the electrical resistance of the product depends not only on the resistivity of the rubber but also on the shape and most probable positions of charge generation and discharge.

Natural rubber is normally considered to be an electrically insulating material but it can be an electrically insulating material but it can be compounded to give electrical resistivity lying anywhere between 1 ohm/cm. and 10^{15} ohm/cm. The most common means of reducing resistance is to add a suitable carbon black (super conductive furnace). Resistance falls with a decrease in particle size, increase in black "structure" and increase in concentration. For light coloured products certain grades of aluminium silicate may be used as antistatic fillers although these are usually less effective in reducing resistance than the super conductive furnace. There are also other proprietary antistatic agents that are available, such as ethylene oxide, but still these agents are less effective then the super conductive furnace.

The applicant has found that carbonised rice husk is particularly suited for use as a filler in plastics as it has been found to enhance the conductivity and flame retardant properties of the composite plastics.

Honeycomb structure of a matrix is supposed to be one of the strongest structures that have been determined by Structural Engineers. The strength comes about from the full depth hexagons and half-depth trapezoids. This type of structures is presently used as designs for building bridge decks.

The rice husk has a similar type of honeycomb design, which results in not only providing strength to the matrix, but also has sound and thermal insulation properties. The Sound insulation property is provided by the micro-cellular structures formed by the honeycomb structure in the brown rice husk. Thus the sound is trapped within the micro-cellular structure. This property is inherent to the brown (fresh) rice husk. The Thermal insulation property is provided by the honeycomb structure, which is strengthened by the sllica and fibre which predominately present in brown rice husk and lesser in the carbonised (depend to the rate of carbonising) rice husk.

The presence of appreciable quantities of oxygen on the surface of carbonised rice husk acts as insulation for each aggregate, thereby reducing the conductivity and also reducing the flammability. The presence of nitrogen and oxygen in the fresh husk not only enables the blowing effect but also nitrogen being inert reduces the flame spread. The volume of gas (nitrogen/oxygen) evolution per gram of fresh rice husk is 240 ml/g. The husk's decomposition temperature is at about 280°C and curing temperature of rubber and ethyl vinyl acetate is between 130°C-180°C, thus when urea (dinitroso pentamethylene tetramine) is milled along the decomposition temperature is reduced within the curing temperatures. The presence of silica in the rice husk provides better mechanical strength.

Typical chemical and physical properties of fresh and carbonised rice husk are detailed as follows:

- consists of 20-23% of paddy
- husk burning: 20% ash by weight
  90-95% is silica (amorphous and crystalline)
- physical characteristics: bulk density 96.12-112.14 kg/m^3
- pH 7.14 (husk ash)
- moisture content 5.6-7.2%, dry basis
- ash 22.2%

Chemical Composition

Moisture Content	: 5.6-7.2%, dry basis
Ash:	: 22.2%
Protein	: 2.4%
Crude fat	: 0.7%
Carbohydrate	: 32.0%

	Fresh RH	**Carbonised RH**
Al_2O_3	0.025%	0.023%
CaO	0.36%	0.12%
NaO	0.034%	0.018%
SiO_2	96.2%	53.88%
Fe_2O_3	0.041%	0.022%
MgO	0.16%	0.078%
K_2O	0.69%	0.95%
P_2O_5	0.57%	0.27%

It is an object according to one aspect of the present invention to provide an alternative filler which will enhance the antistatic, flame retardant, accelerator, plasticiser blowing and/or other physical or mechanical in composite materials. The filler is desirably cheap, environmentally friendly and replenishable and it does not compromise other characteristics of the composite material.

SUMMARY OF INVENTION

The present invention in one aspect resides in the use of carbonised vegetative-based filler to provide improved antistatic, flame retardant, accelerator, plasticiser, blowing and/or other physical or mechanical characteristics in composite materials.

Preferably, the carbonised vegetative-based filler is carbonised rice husk.

Preferably, the carbonised rice husk is burnt at about 800°C for about 4 seconds. Most preferably, the carbonised rice husk is burnt at 803-804°C for 3-4 seconds.

In another aspect the invention resides in a composite material, more particularly a composite plastic including a vegetative-based filler when used as a conductive or flame retardant article.

Preferably, the carbonised vegetative-based filler is carbonised rice husk which has been burnt at 803-804°C for 3-4 seconds.

The present invention also exhibits the usage of fresh and carbonised rice husk as a blowing agent when used in combination with recycled (reclaimed), or virgin natural rubber or other suitable thermoplastic materials. Though other conventional blowing agents have been used with natural or synthetic rubber to achieve the similar products but so far no blowing agents have been used with recycle (reclaim) rubber to produce similar products. Furthermore the conventional blowing agents are expensive and dosages are higher as compared to the fresh rice husk. For example for the conventional blowing agent, the dosage is about 6.5-7 phr, whereas the fresh/carbonised rice husk, the dosage is between 1.5 to 3 phr. When rice husk is used at different dosages the blowing effect is different. It was also noted that the rice husk does not only work as a blowing agent, but also as a plasticizer and a filler. The properties achieved are comparable to conventional blowing agents, when using fresh or carbonised rice, has no difference to the conventional blowing agent other than the colour of the end product.

Ebonite, a hard, dark-coloured plastic-like material, is the reaction product of rubber and a large proportion of sulphur. Simple rubber / sulphur mixtures are seldom used in practice; they suffer from poor processability, require long cure times and lead to excessive shrinkage and heat evolution during cure. Accelerators, fillers, processing aids and other compounding ingredients are widely used in ebonite, as in soft rubber vulcanised rubber, to ease processing, shorten cure times and modify properties. The curing times for ebonite are generally up to ten (10) hours at 150° C, thus making ebonite products expensive. Ebonite can be made from synthetic, such as BR, NBR, SBR and Nitrile rubber and as well as from Natural rubber. High strength, low thermal conductivity, chemical resistance and insulating properties of natural rubber make it a popular choice. Although it has been superseded in many applications by synthetic thermoplastic and thermosets, it is still used for outstanding chemical resistance and electrical properties coupled with high mechanical strength and ease of machining.

The present invention exhibits the usage of fresh and carbonised rice as an accelerator when used in combination with recycled (devulcanised) or virgin natural rubber, and at the same time making ebonite a conductive product when

carbonised rice husk is used. Though other conventional accelerators have been used with natural or synthetic rubber (virgin or recycled) to achieve the similar products but so far no accelerators like the rice husk material have been used with recycled (devulcanised) rubber to produce similar products. Further more the conventional accelerators and conductive carbon black are expensive and difficult to blend and process. When rice husk is used singularly at different dosages the activation effect is different to meet industrial requirements. Generally for ebonite production the sulphur content should be in the range of 25-40 phr, but whereas when fresh rice husk between 25-30 phr is used the sulphur content could be reduced to 20-25 phr. Accelerators are less effective in ebonite than in soft rubber and large quantities are generally required. Basic accelerators such as guanidines and aldehyde-amines are preferred. Inorganic activators such as magnesium oxide, magnesium carbonate and lime are also effective when used with organic accelerators to reduce cure time without the risk of over heating.

Common inorganic fillers used in ebonite are china clay, talc, silica, whiting and magnesium oxide. These also reduce shrinkage and heat evolution but loaded ebonite generally have weaker mechanical properties than unloaded ones. Carbon black does not reinforce ebonite and is normally only added for pigmentation, although conductive carbon black are sometimes used to prepare electrically conducting ebonite.

BEST MODE

Following is an example of the invention, in this example the filler is carbonised rice husk (CRH) which has been burnt at 803-804°C for 3-4 seconds. After this the CRH is obtained.

The manner in which the rice husk is burnt is believed to be important in achieving the desired surface area, surface structure and porosity necessary for conductivity and flame retardant and blowing properties in the composite plastics to be achieved. At this stage the range of temperature and the duration of the time of burning which achieves the desired result has not been fully explored, however it is predicted that the temperature range will be from about 100-950°C and the time range will be from about 2-30 seconds, although these ranges may

be much narrower. The importance of controlled burning in a prescribed time results in obtaining better surface area and porosity which in turn offers ideal properties emitting anti-static, flame retardant and enhancing physical properties of the material. In the absence of controlled burning, the surface area, surface structure and porosity would be distorted. While the present exemplification involves use of carbonised rice husk it is possible that the desired results may be achieved by use of other carbonised vegetative-based fillers.

EXAMPLE 1

A thermoset application called pulforming was used to manufacture fibre reinforced bolts. Fibre glass tows (36 tow of 8000 tex) are pulled into a resin bath that contains:

1. Polyester and Vinyl Ester combination, ie. 60% Vinyl Ester (Derakane 411 - Dow Chemical) and 40% Polyester (Everpol 3260 AR - P.T. Arinde).
2. Zinc Stearate (mould releasing agent) - 1.18% of the resin weight BYK 980 (improves wetting and dispersing of fillers in glass fibre reinforcement compounds) - 1.5% of the filler weight.
3. BYK 515 (air releasing agent) - 0.5% of the total weight of the resin mixture.
4. BYK 996 (wetting and dispersing additive for mineral fillers in hot curing, glass fibre reinforced UP-resin systems) - 2% of the resin weight.
5. Fillers ($Ca_2(CO)_3$ & Carbonised Rice Husk (mesh size 325) @ 55% & 12% of the resin weight).
6. Aluminium Trihydrate (2.4% of the resin weight).
7. Catalyst TBPH (Tertiary Butyl Peroxy-2-Ethyl Hexanoate) - 2.12% of the resin weight.
   TBPB (Tertiary Butyl Perbenzoate) - 0.53% of the resin weight.

The wet fibre glass tow is pulled into the mould and compressed at a pressure of 800psi (5600kPa) for 3.8 minutes at 130°C. Then the bolt is pulled out of the mould and left to cure.

The following day tests were carried out on the bolt with the following results:

Tensile strength at the thread - 50kN

Torque - 45ft/lb

Bond strength - BS 1610:Part 1, Grade 1.0

Fire rating - BS 5865:1980 - Persistence flame shall be less than 10 seconds

Electrical conductivity - less than 10 to the power of 9 Ohms.

EXAMPLE 2

All chemicals used are taken by percentage of weight of rubber. The rubber and the chemicals are mixed in a Banbury, for 5 minutes. Recycled rubber (reclaim) (220g) is first milled with zinc oxide (4.5%) - accelerator, which is followed with stearic acid (1.8%) - activator, Mercadibenzothiazole disulphide (MBTS)(0.5%), Tetramethylthiuram disulphide (TMTD)(0.2%)- accelerator, fresh rice husk (27%)-blowing agent and filler and sulphur (2.7%)- vulcanisate. Then the mixed compound is milled for five (5) minutes to form a sheet that is ready for curing. Then a piece of the sheet weighing about 32g is placed in a mould that it is to be cured for two (2) minutes in a oven at 150°C temperature. The conventional curing time is six (6) minutes at the same temperature of 150°C.

The rubber and the chemicals are mixed in a Banbury, for 5 minutes. The similar approach has been done for using SBR Rubber (100g), silica (62g), Peg 1500 (2.5g), Paraffin oil (5g), Zinc oxide (2.5g), Wing stay (0.5g), Wax (1g), Mercadibenzothiazole disulphide (MBTS) (1.5g), Tetramethylthiuram disulphide (TMTD) (0.2g), Stearic acid (1.5g) and Sulphur (2g). The milling was done for ten (10) minutes and later cured in the oven for six (6) minutes at 150°C.

This exercise was repeated by using fifty (50) percent of the virgin material compound and fifty (50) percent recycled (reclaimed) material compound, and cured in the oven at 150°C for two (2) minutes.

With the level, of rice husk dosage, the blowing effect can be controlled to suit the industry's requirement.

Machine Operating Conditions

Virgin Rubber:

PROPERTIES	SMR-10	TYRE DUST	TYRE DUST WITH BROWN RICE HUSK	EXAMPLE 1 WITH BROWN RICK HUSK	EXAMPLE 1 WITHOUT BROWN RICE HUSK
Mooney Viscosity MLI + 3, 100°C	60	36.6	31.8	24	30
Monsanto Rheometer, 150°C					
Scorch time	1.4	3.8	1.2	2	2.5
Cure time	7.4	3.5	3.25	4	4

By using rice husk the Mooney viscosity was lower than the conventional filler, thus lowering scotch time (time taken by the rubber compound to flow into the mould) and curing time (time taken to cure rubber compound) respectively. As such this leads to a cheaper production system. Presently various fillers and blowing agents are being used in the production of soft/spongy rubber that would produce different types of cell structures for an end product, but the cost determines the market.

EXAMPLE 3

All chemicals used are taken by percentage of weight of rubber. The rubber and the chemicals are mixed in a Banbury, for six (6) minutes. The recycle (devulcanised) rubber is first milled with magnesium oxide (2%) - accelerator, which is followed with Diphenylguanidine (2%) - accelerator, fresh rice husk (30%) - accelerator and filler and sulphur (30%) - vulcanisate. After the milling at the Banbury for ten (10) minutes, it is then milled into a sheet. The mould was heated in the oven press to 150°C then the sheeted rubber is placed in the mould and it is cured for twelve (12) minutes. The conventional curing time is between eight to ten hours at the same temperature of 150°C.

A conventional formula for ebonite was selected to compare. The rubber and the chemicals are mixed in a Banbury, for 5 minutes. The similar mixing as above was followed, using SBR 5 Rubber (100g), ebonite dust (100g), China clay (50g), Magnesium oxide (5g), Diphenylguanidine (3g), Linseed oil (5g) and Sulphur (45g). The milling was done for ten (10) minutes and later cured in the oven for eight (8) hours at 150°C.

Mix properties; Rice Husk filled mix

Mooney viscosity, MLI + 3, 100°C	24
Mooney viscosity, MLI + 3, 120°C	18.5
Mooney scorch, t_5, MLI + 3, 120°C min.	5.8
Monsanto Rheometer, 160°C	
time to 95% cross-linking, s	110

By using rice husk the curing time is reduced tremendously twelve minutes as compared to eight to ten hours. The sulphur content in the rubber polymer is reduced by fifteen percent.

The results are based on cure time, the formulation with rice husk cures faster than the formulation without rice husk, i.e. twelve minutes for with brown rice husk and about eight hour without rice husk.

EXAMPLE 4

All chemicals used are taken by percentage of weight of natural rubber (NR). The natural rubber and the chemicals are mixed in an open mill or kinder, for six (6) minutes. Natural rubber is first milled with stearic acid (1%) and zinc oxide (5%) activator, which is followed with rice husk (blowing agent) (2.5-3.5%), calcium carbonate - (40%), promoter - urea based (2.5-3.5%), silica (10%), accelerator dibenzthiazyldisulphide (MBTS) (0.05%) and catalyst sulphur (1.5%). After the milling at the open mill or kinder for ten (10) minutes, it is then milled into a sheet. The mould was heated in the oven press to 160°C then the sheeted natural rubber is placed in the mould and it is cured for twenty-two (22) minutes.

The temperature for curing could be from 145°-160°C and the cure time may differ according to the mould size.

Cured properties; rice husk filled blowed mix - micro-cellular cells.

1.	Hardness	Askar C	35
2.	Shrinkage	%	5
3.	Specific Gravity	g/cc	0.3-0.35

By using rice husk as a blowing agent the catalyst percentage could be reduced and as well as the percentage of blowing agent used.

EXAMPLE 5

THERMOPLASTIC (EVA)

All chemicals used are taken by percentage of weight of thermoplastic - Ethyl Vinyl Acetate (EVA). The EVA and the chemicals are mixed in an Open Mill or Knider, for six (6) minutes. Ethyl Vinyl Acetate (EVA) is first milled with Stearic Acid (1%) and zinc oxide (5%) accelerator, which is followed with Rice Husk (Blowing agent) (2.5%). Magnesium carbonate - (10%), Promoter - urea based (5%) and catalyst Diacyl Peroxide (1%). After the milling at the Open mill or Knider for ten (10) minutes, it is then milled into a sheet. The mould was heated in the oven press to 160°C then the sheeted EVA is placed in the mould and it is cured for twenty-two (22) minutes.

The temperature for curing could be from 145°-160°C and the cure time may differ according to the mould size.

Cured properties: Rice Husk filled blowed mix - micro-cellular cells

1.	HARDNESS	Askar C	29-35
2.	SHRINKAGE	%	2
3.	SPECIFIC GRAVITY	g/cc	0.200
4.	COMPRESSION SET	%	80

By using rice husk as a blowing agent the catalyst percentage could be reduced and as well as the percentage of blowing agent used.

EXAMPLE 6

The rice husk is mixed by weight with tyre crumbs(35-40 mesh) and an effluent from the palm oil mill called Scavenger (which have a fatty acid content (C_8-C_{18}). From literature it has been reported that by using tyre crumb with the binder (bitumen) there is an improve of properties for the asphalt mixture. This invention not only uses tyre crumb along with rice husk and an oil palm effluent to further improve the properties. As well as the formulation address the recyclability of all agro waste by-products to be used in the road surfacing industries. The formulation of the rice husk mixture as follows:

Rice husk	50%
Tyre crumb	45%
Scavenger	5%

In this particular example the usage of rice husk mixture is divided into two categories:

A. RICE HUSK MIXTURE USED IN MODIFIED BINDER

B. RICE HUSK MIXTURE USED IN AS FILLER

A. The rice husk mixture is added to the bitumen first in compliance to the SOCIETY OF HIGHWAY PROCEDURE (SHRP) to manufacture modified bitumen. The bitumen is first heated to about 160°C, then the rice husk mixture twenty percent 20% by weight of bitumen is mixed with the heated bitumen for about one hour. As a result of this reaction a modified bitumen is made. From here 5-7% by weight of this modified bitumen is added to the aggregate. The aggregate is first heated to about 200°C and the modified bitumen is mixed for three to four minutes. The modified bitumen with rice husk mixture complies to all requirement of the SHRP.

B. The rice husk mixture is added as a filler to the aggregate, by four (4%) by weight to the aggregate weight. The aggregate is first heated to 200°C, and is allowed to cool to about 160°C, then the rice husk mixture is added and mixed and lastly the bitumen 5-6% by weight of aggregate is added and mixed for 3-4 minutes. This blending with rice husk mixture complies to all requirement of the Marshall Properties.

TABLE 1

PROPERTIES OF RICE HUSK MODIFIED BINDER - SHRP

SHRP	80/100	RICE HUSK MIXTURE
FLASH POINT TEMP. °C	230	240
SOFTENING POINT, °C	44-50	55-70
PENETRATION @ °C 25, dmm	80-100	60-100
BROOKFIELD VISCOSITY @ 135°C, MPaS	<500	>1500
DYNAMIC SHEAR RHEOMETER		
PG 70		
ORIGINAL		
G* (Pa)	<1000	>1200
δ(°)	>80	>80
G*/Sin δ	<1000	>1200
AFTER RTFOT		
G* (Pa)	<1000	>3800
δ(°)	>80	>70
G*/Sin δ	<1000	>3800
AFTER PAV		
G* (Pa)	<1000	>230
δ(°)	>80	>50
G*/Sin δ	<1000	>260
PG 76		
ORIGINAL		
G* (Pa)	<1000	>1800
δ(°)	>80	>70
G*/Sin δ	<1000	>1800
AFTER RTFOT		
G* (Pa)	<1000	>2400
δ(°)	>80	>70
G*/Sin δ	<1000	>2600
AFTER PAV		
G* (Pa)	<1000	>230
δ(°)	>80	>50
G*/Sin δ	<1000	>280

TABLE 2

MIXED PROPERTIES OF RICE HUSK MODIFIED BINDER

PROPERTIES	80/100	RICE HUSK MIXTURE
MARSHALL STABILITY (kN)	5-10	>13
FLOW (mm)	2-4	2-4
QUOTIENT (kN/mm)	1-3.5	3-4
RESILIENT MODULUS @ 25°	>2000	>2800

TABLE 3

MIXED PROPERTIES OF RICE HUSK MIXTURE AS FILLER

PROPERTIES	80/100	RICE HUSK MIXTURE
MARSHALL STABILITY (kN)	6-10	>12
FLOW (mm)	2-4	2-4
QUOTIENT (kN/mm)	1-3.5	3-4
RESILIENT MODULUS @ 25°	>2000	>2800

TABLE 4

PREFERRED PARTICLE SIZE AND DOSAGE OF FRESH AND/OR CARBONISED RICE HUSK FOR PARTICULAR COMPOSITE MATERIALS

COMPOSITE MATERIAL	FRESH/DOSAGE	CARBONISED/DOSAGE
BITUMEN (MECHANICAL PROPERTY	100 MESH - 40-60 phr	-
THERMOPLASTIC (EVA) BLOWING CHARACTER	325-400 MESH - 1.5-2.5 phr	325-400 MESH - 1.5-2.6 phr
THERMOPLASTIC (EVA) MECHANICAL PROPERTY	325-400 MESH - 1.5-5 phr	325-400 MESH - 1.5-2.5 phr
RUBBER (N.R./S.R.) BLOWING CHARACTER	325-400 MESH - 1.5-27 phr	325-400 MESH - 1.5-27 phr
EBONITE N.R. (REDUCE CURE TIME)	100-200 MESH 18-30 phr	-
RUBBER (N.R./S.R.) SCOTCH TIME	100-200 MESH 5-10 phr	100-200 MESH 5-10 phr
THERMOSET RESIN (FLAME PROPERTY)	-	325 MESH 10-15 phr
THERMOSET RESIN (MECHANICAL PROPERTY)	100-200 MESH 10-15 phr	100-200 MESH 10-15 phr
THERMOSET RESIN (ANTISTATIC)	-	325 MESH 10-15 phr
RUBBER-LATEX (N.R./S.R.) ANTISTATIC	-	325-400 MESH 5-15 phr
RUBBER (N.R./S.R.) ANTISTATIC	-	325-400 MESH 5-15 phr
CONCRETE (MECHANICAL PROPERTY)	100-200 MESH 10-15 phr	100-200 MESH 10-15 phr

N.R. - NATURAL RUBBER
S.R. - SYNTHETIC RUBBER

It will of course be realised that whilst the above has been given by way of illustrative examples of this invention, all such and other modifications and variations hereto, as would be apparent to persons skilled in the art, are deemed to fall within the broad scope and ambit of this invention as herein set forth. For instance, while the preceding examples relate to the use of fresh and/or carbonised rice husk it would be apparent to a person skilled in the art that other cereal husks such as sorghum husk may be suitable.

Throughout the description and claims of the specification where reference is made to the dosage of fresh and/or carbonised rice husk, this dosage is expressed in terms of "phr" (parts per hundred) based on the weight of the composite material into which the rice husk is being introduced.

Throughout the description and claims of the specification the word "comprise" and variations of the word, such as "comprising" and "comprises", is not intended to exclude other additives, components, integers or steps.

THE CLAIMS DEFINING THIS INVENTION ARE AS FOLLOWS:

1. A filler for use in composite materials wherein said filler comprises carbonized vegetative-based material wherein said carbonized vegetative-based material is the product of burning fresh vegetative-based material at 803° to 804°C for 3 to 4 seconds.

2. A filler according to claim 1 wherein the carbonized vegetative-based material is carbonized rice husk.

3. A process for the production of a carbonised vegetative-based filler wherein said process comprises burning a fresh vegetative-based material at about 803° to 804°C for 3 to 4 seconds.

4. A process according to claim 3 wherein the fresh vegetative material is ground to a particle size of from 100 mesh to 400 mesh.

5. A process according to claim 3 or claim 4 wherein said process utilises fresh rice husk as the vegetative material.

6. A filler according to any one of claims 1 or 2 when produced by a process according to claim 4.

7. A method for improving the anti-static, flame retardant, accelerator, plasticiser and/or blowing characteristics of a composite material wherein said method comprises blending into the composite material with a carbonised vegetative-based filler according to claim 1 or claim 2 and wherein said blending is substantially completed prior to incorporation of any additives, if any.

8. A method according to claim 7 wherein the carbonised vegetative filler has a particle size of from 100 mesh to 400 mesh.

9. A method according to claim 7 or claim 8 wherein the composite material is latex (NR/SR) the dosage of the carbonised vegetative filler is from 1.5 to 2.5 phr (parts per hundred).

10. A method according to any one of claims 7 to 9 wherein said composite material is selected from the group comprising:

i) thermoplastic resins;
ii) thermoset plastics;
iii) rubbers and elastomeric materials;
iv) conductive coatings;
v) printing inks;
vi) bitumen; and
vii) concrete.

11. A composite material having improved anti-static, flame retardant, accelerator, plasticiser and/or blowing characteristics wherein said composite material is produced by the method of any one of claims 7 to 10

12. A method for improving the mechanical properties of bitumen, said method comprising blending fresh and/or carbonised rice husk together with tyre crumb into said bitumen.

13. A method according to claim 12 wherein the rice husk has a particle size of from 100 to 200 mesh and the dosage of rice husk is between 40 to 60 phr.

14. A method according to claim 12 or claim 13 wherein the rice husk and tyre crumb is mixed in composition with a palm oil effluent prior to it being added to the bitumen.

15. A method according to claim 14 wherein the composition comprises about 50% tyre crumb, about 45% rice husk and about 5% palm oil effluent.

16. A method according to claim 14 or claim 15 wherein the composition is added in a dosage amount of about 20% by weight of the said bitumen.

17. A method for improving the blowing character of a thermoplastic resin this method comprising blending fresh and/or carbonised rice husk into said thermoplastic resin. wherein the rice husk has a particle size of between 325 to 400 mesh and the dosage of the rice husk is between 1.5 to 2.5 phr.

18. A method for improving the mechanical properties of thermoplastic resin including compression strength, said method comprising blending rice husk into said thermoplastic resin wherein said rice husk has a particle size of between 325 to 400 mesh and the dosage of the rice husk is between 1.5 and 2.5 phr.

19. A method for improving the blowing character of rubber said method comprising blending fresh and/or carbonised rice husk into said rubber.

20. A method according to claim 19 wherein the rice husk has a particle size of between 325 to 400 mesh and the dosage of the rice husk is between 1.5 and 27 phr.

21. A method for reducing the cure time of ebonite NR wherein said method comprises blending fresh rice husk into said ebonite NR.wherein the rice husk has a particle size of between 100 to 200 mesh and the dosage of the rice husk is between 16 to 30 phr.

22. A method for improving the scotch time of rubber said method comprising blending fresh and/or carbonised rice husk into said rubber.

23. A method according to claim 22 wherein the rice husk has a particle size of between 100 to 200 mesh and the dosage of rice husk is between 5 to 10 phr.

24. A method for improving the flame retardant ability of a thermoset resin said method comprising blending carbonised rice husk according to claim 2 into said thermoset resin.

25. A method according to claim 24 wherein the carbonised rice husk has a particle size of 325 to 400 mesh and the dosage of carbonised rice husk is between 10 to 15 phr.

26. A method for improving the mechanical properties of thermoset resins including tensile and torque strength, said method comprising blending carbonised rice husk according to claim 2 into said thermoset resin.

27. A method according to claim 26 wherein the rice husk has a particle size of between 100 to 200 mesh and the dosage of rice husk is between 10 to 15 phr.

28. A method for improving the anti-static properties of a thermoset resin said method comprising blending carbonised rice husk according to claim 2 into said thermoset resin.

29. A method according to claim 27 wherein the carbonised rice husk has a particle size of between 325 to 400 mesh and the dosage of carbonised rice husk is between 10 to 15 phr.

30. A method for improving the anti-static properties of rubber and/or rubber latex said method comprising blending carbonised rice husk according to claim 2 into said rubber and/or rubber-latex.

31. A method according to claim 30 wherein the carbonised rice husk has a particle size of between 325 to 400 mesh and the dosage of carbonised rice husk is between 5 to 15 phr.

32. A method for improving the mechanical properties of concrete said method comprising blending carbonised rice husk according to claim 2 into said concrete.

33. A method according to claim 32 wherein the rice husk has a particle size of between 100 to 200 mesh and the dosage of the rice husk is between 10 to 15 phr.

34. A filler according to claim 1 substantially as hereinbefore described with reference to any of the examples.

35. A process according to claim 3 substantially as hereinbefore described with reference to any of the examples.

36. A method according to claim 7 substantially as hereinbefore described with reference to any of the examples.

37. A composite material according to claim 11 substantially as hereinbefore described with reference to any of the examples.

DATED: 17 November, 2000

PHILLIPS ORMONDE & FITZPATRICK
Attorneys for:

Contract Research & Development (M) Sdn. Bhd.
And Mr. D J .Tadgell

PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 7 : C09K — A2

(11) International Publication Number: WO 00/42116

(43) International Publication Date: 20 July 2000 (20.07.00)

(21) International Application Number: PCT/AU00/00018

(22) International Filing Date: 17 January 2000 (17.01.00)

(30) Priority Data:
PP 8198 — 18 January 1999 (18.01.99) — AU

(71) Applicants *(for all designated States except US)*: CONTRACT RESEARCH & DEVELOPMENT (M) SDN. BHD. [MY/MY]; Suite 101, 1st floor, Wisma Kwang Tung Association Building, 44 Jalan Pudu, 55100 Kuala Lumpur (MY). TADGELL, David, John [AU/AU]; 367 Collins Street, Melbourne, Victoria 3000 (AU).

(72) Inventors; and

(75) Inventors/Applicants *(for US only)*: SIVASITHAMBARAM PILLAI MAILVAGANAM PILLAI, Mailvaganam, Thavalingam [MY/MY]; (MY). HAJI AHMAD, Khalid [MY/MY]; (MY). VETTIVALOO ARUNASALAM, Arulgnanam [MY/MY]; Suite 101, 1st floor, Wisma Kwang Tung Association Building, 44 Jalan Pudu, 55100 Kuala Lumpur (MY).

(74) Agent: PHILLIPS ORMONDE & FITZPATRICK; 367 Collins Street, Melbourne, Victoria 3000 (AU).

(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published

Without international search report and to be republished upon receipt of that report.

(54) Title: USE OF VEGETATIVE MATERIAL AS A FILLER IN COMPOSITE MATERIALS

(57) Abstract

The present invention provides a filler for use in composite materials wherein said filler comprises a vegetative–based material and wherein said vegetative–based material can be fresh or carbonised. In one particularly preferred embodiment the present invention utilises carbonised rice husk. In a further aspect of the present invention there is provided a process for the production of a carbonised vegetative–based filler wherein said process comprises burning a fresh vegetative–based material at about 800 °C for about 4 seconds.

UNITED STATES OF AMERICA DECLARATION FF34086/01

(REV. 2-11-94) Docket No. P0557/7045 DRW

DECLARATION FOR PATENT APPLICATION

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled USE OF VEGETATIVE MATERIAL AS A FILLER IN COMPOSITE MATERIALS the specification of which

(check one)

☐ is attached hereto
☐ was filed on _____, as Application Ser. No. _____ amended on_____ (if applicable).

☒ was filed as a PCT international application No. PCT/AU00/00018, on 17 January 2000 and was amended under PCT Article 19 on _____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign PCT Application(s) and any priority claims under 35 U.S.C. §119:			Priority Claimed
PP8198	AU	18 January 1999	
(Number)	(Country if PCT so indicate)	(DD/MM/YY Filed)	YES ~~NO~~
PCT/AU00/00018	PCT	17 January 2000	
(Number)	(Country)	(DD/MM/YY Filed)	YES ~~NO~~
(Number)	(Country)	(DD/MM/YY Filed)	YES NO

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) or PCT international application(s) designating the United States of America listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

(Application Serial No.)	(filing date)	(status-patented, pending, abandoned)
(Application Serial No.)	(filing date)	(status-patented, pending, abandoned)

PCT Applications designating the United States:

(PCT Appl. No.)	(U.S. Ser. No.)	(PCT filing date)	(status-patented,pending,abandoned)

I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

M. Lawrence Oliverio, Reg. No. 30,915 Therese A. Hendricks, Reg. No. ~~30,389~~

Address all telephone calls to Therese A. Hendricks at telephone no. (617) 720-3500. Address all correspondence to

Therese A. Hendricks
c/o Wolf, Greenfield & Sacks, P.C.,
~~Federal Reserve Plaza~~
600 ~~Atlantic Avenue~~
Boston, MA 02210-2211

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Inventor's signature _____ 31 July 2001 Date

Full name of sole or first inventor MAILVAGANAM THAVALINGAM SIVASITHAMBARAM PILLAI
Citizenship MAILVAGANAM PILLAI
Residence MALAYSIAN
Post Office Address Same Suite 101, 1st Floor, Wisma Kwang Tung Association Building, 44 Jalan Pudu, 55100, Kuala Lumpur, Malaysia

Inventor's signature _____ 31 July 2001 Date

Full name of joint inventor KHALID ~~HAJI~~ AHMAD
Citizenship MALAYSIAN
Residence 1st Floor, Wisma Kwang Tung Association Building, 44 Jalan Pudu,
Post Office Address Same 55100, Kuala Lumpur, Malaysia

Inventor's signature _____ 31 July 2001 Date

Full name of joint inventor ARULGNANAM VETTIVALOO ARUNASALAM
Citizenship MALAYSIAN
Residence Suite 101, 1st Floor, Wisma Kwang Tung Association Building,
Post Office Address Same 44 Jalan Pudu, 55100, Kuala Lumpur, Malaysia